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Title:

NOVEL LACTITOL MONOHYDRATE CRYSTALS AND HONEY-CONTAINING CRYSTALS COMPRISING THE SAME AS WELL AS PROCESSES FOR PRODUCTION THEREOF

Claim(s):

Honey = melam

1. Lactitol monohydrate crystals represented by molecular weight of $C_{12}H_{24}O_{11}.H_2O$ and having a melting point of 102 to 105 °C.

2. Honey-containing crystals comprising lactitol monohydrate crystals represented by molecular weight of $C_{12}H_{24}O_{11}.H_2O$ and having a melting point of 102 to 105 °C.

3. Lactitol monohydrate crystals as claimed in claim 1, wherein said lactitol monohydrate crystals have a lactitol purity of at least 95 wt% per solid content.

4. Honey-containing crystals comprising lactitol monohydrate crystals as claimed in claim 2, wherein said lactitol monohydrate crystals have a lactitol purity of at least 95 wt%

per solid content.

5. Lactitol monohydrate crystals as claimed in claim 1, wherein said lactitol monohydrate crystals are a composition.

6. Honey-containing crystals comprising lactitol monohydrate crystals as claimed in claim 2, wherein said honey-containing crystals comprising lactitol monohydrate crystals are a composition.

7. Lactitol monohydrate crystals as claimed in claim 1, wherein said lactitol monohydrate crystals are molded.

8. Honey-containing crystals comprising lactitol monohydrate crystals as claimed in claim 2, wherein said honey-containing crystals comprising lactitol monohydrate crystals are molded.

9. A process for producing lactitol monohydrate crystals which comprises crystallizing from a lactitol aqueous solution lactitol monohydrate crystals represented by molecular weight of $C_{12}H_{24}O_{11}\cdot H_2O$ and having a melting point of 102 to 105°C, and collecting the crystals.

10. A process for producing honey-containing crystals comprising lactitol monohydrate crystals which comprises crystallizing from a lactitol aqueous solution lactitol monohydrate crystals represented by molecular weight of $C_{12}H_{24}O_{11}\cdot H_2O$ and having a melting point of 102 to 105°C, and collecting or solidifying the crystals.

11. A process for producing lactitol monohydrate crystals as claimed in claim 9, wherein a lactitol purity in said lactitol solution is at least 95 wt% per solid content.

12. A process for producing honey-containing crystals comprising lactitol monohydrate crystals as claimed in claim 10, wherein a lactitol purity in said lactitol solution is at least 95 wt% per solid content.

13. A process for producing lactitol monohydrate crystals as claimed in claim 9 or 11, wherein a crystallizing temperature is 20 to 70 °C.

14. A process for producing honey-containing crystals comprising lactitol monohydrate crystals as claimed in claim 10 or 12, wherein a crystallizing temperature is 20 to 70 °C.

15. A process for producing lactitol monohydrate crystals as claimed in claim 9, 11 or 13, wherein seed crystals are copresent in said lactitol solution.

16. A process for producing honey-containing crystals comprising lactitol monohydrate crystals as claimed in claim 10, 12 or 14, wherein seed crystals are copresent in said lactitol solution.

DETAILED DESCRIPTION OF THE INVENTION

[Field of the Invention]

The present invention relates to novel lactitol monohydrate crystals, and honey-containing crystals comprising the same as well as processes for production thereof.

[Prior Art]

Lactitol is 4-ß-D-galactopyranosyl-D-sorbitol and has a structure that the glucose moiety of lactose is reduced to sorbitol.

Conventionally known lactitol and a process for production

thereof are described in, for example, J. Agricultural and Food Chemistry, 27, 4 (1979), 680-686 and the process comprises using a lactose aqueous solution having a concentration of 30 to 40 wt% as a starting material, hydrogenating the aqueous solution at a temperature of 100 °C under hydrogen pressure of 40 atms using Raney nickel, then precipitating the catalyst and filtering and removing the same, and purifying lactitol with ion exchange resin, activated carbon, etc.

When compared with sweetness of 5% sucrose aqueous solution as 100, lactitol thus obtained has relative sweetness of 36% in the same concentration, showing that its sweetness is lower than that of sorbitol (relative sweetness of 65%) and xylitol (relative sweetness of 96%).

On the other hand, decomposition rate when lactitol is hydrolyzed with α-glucosidase (maltase) as in West German Patent (Meizena, 1974) is markedly slower than hydrolysis rate of lactose or maltose.

For example, lactose is almost completely hydrolyzed by ß-galactosidase in 45 minutes, but 10 to 15% of lactitol is merely hydrolyzed in 45 minutes.

Accordingly, lactitol is digested and absorbed only with difficulty in the digestive tract and also fermented with stomatic bacteria only with difficulty. Therefore, lactitol is suitable for patients with diabetes, obese subjects and people who care adult diseases or caries, as sweet sources for low calorie food, diet food, low caries food, healthy food, etc.

Since lactitol has a low hygroscopic property as compared to

sorbitol, glycerol, xylytol, etc. and its aldehyde group is reduced, lactitol is stable to heat or an alkali. Thus, lactitol can be advantageously utilized in various foodstuffs.

Lactitol crystals are described in some publications. For example, in the reports published in Comptes Rendus Hebdomadaires des Séances de l'Académie des Sciences, 170, 47-50 (1920) and J. Am. Chem. Soc., 74, 1105 (1952), mention may be made on lactitol dihydrate crystals having a melting point of 76-78 °C and a specific rotary power of +12.2 ° and anhydrous lactitol crystals having a melting point of 146 °C and a specific rotary power of +14 °.

Lactitol monohydrate crystals are also mentioned in (1) J. Agricultural and Food Chemistry, 27, 4 (1979), 680-686 and (2) Japanese Patent Application Laid-Open No. 85900/83. In the Japanese patent application, there are mentioned (i) lactitol crystals having a melting point of 94-97 °C and containing 1% of lactulitol (4-ß-D-galactosyl-D-mannitol) and 3% of mannitol and (ii) crystals having a melting point of 121-123 °C, having a calculated density of crystals of 1.528 g/cm^2, containing 4 lactitol molecule and 4 water molecule in the unit lattice, having a unit lattice size of a-7.808 Å, b-12.685 Å and c-15.931 Å, and having a space group being $P2_12_12_1$, at page 5, left lower column, line 10 to page 6, left upper column, line 4.

[Problems to be solved by the Invention]

However, conventional lactitol anhydride, lactitol dihydrate crystals and lactitol monohydrate crystals as well as processes for production thereof involve various disadvantages in process

and utilization. It has been desired to improve the disadvantages.

That is, conventional lactitol anhydride involves disadvantages that: (a) a relatively difficult drying step is required and hence, there is a problem in the process that much time is taken, (b) there is a problem in the property that the anhydride is relatively strongly hygroscopic so that when packing materials or containers made of anhydrous maltitol crystals or other similar sugar alcohols are used, the crystals absorb moisture in the air and the quality is deteriorated with the passage of time, (c) there is a problem in the utilization that the melting point is high and therefore, in order to use the same as a dispersing medium for other powdery materials, it is necessary to heat to a high temperature so that other components might be decomposed in some occasion; etc. Lactitol dihydrate crystals involve disadvantages that the crystals have a melting point as low as 76-78 °C and cannot be used as powders in the field of food in which heating at about 100 °C is often required and therefore, their use is limited, etc.

It was expected that lactitol monohydrate would solve these problems. However, conventionally known lactitol monohydrate crystals involve the following problems: lactitol monohydrate crystals described in (i) above contain impurities such as mannitol or lactulitol, etc. and hence, taste bitter so that they are not suitable for food; because of these impurities, the crystals have a low melting point and are not sufficient to construct lactitol monohydrate single crystals. Furthermore, the

crystals described in (ii) above is disadvantageous in process since the process involves so many difficulties that could not be even produced or confirmed, even though the crystals were attempted to produce and confirm the property by way of experiment.

From the foregoing circumstances, it has been desired to improve the various problems on lactitol crystals as described above.

[Means for solving the Problems]

In order to improve the various problems on conventional lactitol crystals, the present inventors have made extensive investigations on the physicochemical properties of lactitol and continued the investigations to survey lactitol crystals having excellent properties.

As a result, by crystallizing from a lactitol aqueous solution, lactitol monohydrate crystals which have not been recited in publications and have excellent properties in their application, and honey-containing crystals comprising the same as well as processes for production thereof have been found. Thus, it has been succeeded in advantageously preparing the lactitol monohydrate crystals and honey-containing crystals containing the same in an industrial scale. The present invention has thus come to be accomplished.

That is, the present invention is directed to lactitol monohydrate crystals represented by molecular weight of $C_{12}H_{24}O_{11}\cdot H_2O$ and having a melting point of 102 to 105°C, and honey-containing crystals containing the same as well as

processes for production thereof.

Physicochemical properties of the lactitol monohydrate crystals of the present invention are described below.

(1) Elemental analysis

Found C = 39.6% H = 7.3% O = 53.1%

Calcd. C = 39.8% H = 7.2% O = 53.0%

(2) Molecular weight

362.3

(3) Melting point

102.0 - 105.4 °C (sample was put on a plate heated at a definite temperature and measured visually)

(4) Differential scanning calorimetry

Maximum endotherm = 100 - 108 °C [measured at an elevation rate of 10 °C/min using a differential scanning calorimeter]

(5) Specific rotary power

$[\alpha]_D^{20}$ = +14.1° (containing 0.1 g in 1 cc of water)

(6) UV absorption

When measured in an aqueous solution, any characteristic absorption is not shown.

(7) IR absorption spectrum

Lactitol monohydrate crystal powders, 10 mg, were mixed with 440 mg of dry KBr. The mixture was mixed to prepare a transparent tablet (about 0.6 mm in thickness) and IR absorption spectrum was measured. The results are shown in Table 1.

Table 1. Wavelength at which IR absorption was observed

3100 - 3400 (nm)
2840 - 2920
1300 - 1430
990 - 1120

(8) Solubility

Solubility of lactitol monohydrate crystals in water was about 55% at 25°C.

(9) Physical properties and color of substance

The crystals are colorless transparent. Fine crystals are white powdery, have sweetness of 35 to 40% of sugar and are odorless. They are not deliquescent. Even during storage at a temperature of 30°C and a relative humidity of 72% for 284 hours, weight increment is merely within an error and hygroscopicity is hardly observed.

When dry weight is measured at 60°C for 72 hours under normal pressure, about 5% decrease in the dry weight is observed.

(10) Solubility in various chemicals

Readily soluble in water, 0.1 N NaOH and 0.1 N HCl; soluble in methanol; sparingly soluble in ethanol; insoluble in chloroform, ethyl acetate.

(11) X ray analysis on crystalline structure

Using single crystal of the novel lactitol monohydrate crystals of the present invention, X ray analysis on crystalline structure was performed. The results reveal that the crystals fall under the rhombic system and the unit lattice contains 4

lactitol molecules and 4 water molecules. A size of the unit lattice is a-7.808 Å, b-12.685 Å and c-15.931 Å. A calculated density of crystals is 1.528 g/cm^2, and its space group is $P2_12_12_1$. The structure of the crystals is shown in Fig. 1.

From the foregoing measurement results, it is assumed that the lactitol monohydrate crystals of the present invention and the honey-containing crystals containing the same are novel lactitol monohydrate crystals which are obviously different from those described in the publications.

Hereinafter processes for producing the lactitol monohydrate crystals of the present invention and the honey-containing crystals containing the same are described below.

The lactitol solution for crystallization which is used in the present invention may be any lactitol solution so long as the lactitol monohydrate crystals of the present invention are crystallized, regardless of processes for producing lactitol.

More specifically, lactitol having a purity of 95% or more is made an aqueous solution having a concentration of 60 to 95 wt%. The temperature is desirably in a range of 0 to 95 °C at which the solution is not frozen and heat loss in the processes is relatively small.

In order to control supersaturation and viscosity of the solution, an organic solvent such as ethanol, acetone, etc. may also be copresent.

For crystallization of the lactitol monohydrate crystals of the present invention, seed crystals preferably having a solid content of 0.01 to 20 wt% based on the solution are added at a

temperature of 20 to 60 °C to the lactitol aqueous solution preferably having a concentration of 60 to 95 wt%. While slowly stirring, the system is cooled to prepare a mass kit.

As described above, the lactitol monohydrate crystals of the present invention or the honey-containing crystals containing the same can be relatively easily crystallized by adding as seed crystals the lactitol monohydrate crystals or the honey-containing crystals containing the same to the supersaturated lactitol solution.

For preparing the lactitol monohydrate crystals or the honey-containing crystals containing the same from the crystallized mass kit, any processes may be applicable so long as the desired product of the present invention can be harvested. Where the moisture content in the crystallized mass kit is small, it is possible to solidify as it is. Where the moisture content is large, it may be possible to collect the lactitol monohydrate crystals.

As the processes, there may be adopted known methods such as various methods for incorporating honey or methods for separating honey, for example, a kneading method, a block grinding method, a fluidizing granulation method, a spray drying method, etc.

Specifically, for example, the method for separating honey comprises separating the mass kit into lactitol monohydrate and honey from by a centrifuging machine. The method makes it easy to the effect that if necessary, the crystals may be washed with a small quantity of water by spraying chilled water. Therefore, the method is suitable for preparing the lactitol monohydrate

crystals having a melting point of 102 to 105 °C in a higher purity.

According to the other methods for kneading, block grinding, fluidizing granulation and spray drying, trace amounts of known lactitol dihydrate crystals or other sugar alcohol, e.g., lactulitol, mannitol, sorbitol, etc. may be sometimes contained as honey components, in addition to the lactitol monohydrate crystals of the present invention, since honey is not separated in these methods.

The kneading method which is one of the method for incorporating honey comprises gradually cooling a lactitol solution having a water content of 5 to 25%, preferably 6 to 20% in a kneader, mixing then after adding seed crystals having a solid content of 0.5 to 80 wt% based on the solution at a temperature lower than the melting point of lactitol monohydrate, preferably at 30 to 70 °C or without adding, and molding the mixture into various shapes, e.g., powders, granules, spheres, rods, plates, cubes, etc., whereby the honey-containing crystals containing the lactitol monohydrate crystals can be obtained in the form or powders or molded forms.

The lactitol monohydrate crystals of the present invention or the honey-containing crystals containing the same have excellent properties as compared to conventionally known lactitol anhydride or lactitol dihydrate crystals. For example, when compared with lactitol anhydride, the crystals of the present invention are advantageous in that the melting point is appropriately low so that drying can be made relatively easily;

the crystals of the present invention are hardly hygroscopic so that it is possible to use relatively cheap packing materials. It is also advantageous, when compared with the lactitol dihydrate crystals, that the melting point is appropriately high and the crystals are suited for use in food cooked at about 100 °C. Accordingly, the crystals of the present invention can be advantageously used for use in food, cosmetics, drugs, raw materials in chemistry, etc.

The lactitol monohydrate crystals of the present invention may be used, if necessary, by mixing with a suitable quantity of one or more other sweeteners such as powder candy, glucose, maltose, isomerized sugar, sugar, molasses, maple sugar, sorbitol, dihydrochalcone, stebioside, α-glycosylstebioside, glycylrrhyzin, saccharin, aspartem, glycine, alanine, etc.

The lactitol monohydrate crystals may also be used by mixing with a thickener such a dextrin, starch, polydextrose, lactose, etc.

In addition, among the lactitol monohydrate crystals of the present invention and the honey-containing crystals containing the same, those in a powdery form may be freely used as they are or, if necessary, by mixing with a thickener, an excipient, a binder, a disintegrator, etc. and preparing into granules, spheres, tablets, rods, plates, cubes, etc.

Furthermore, the lactitol monohydrate crystals of the present invention and the honey-containing crystals containing the same can reduce calorie of beverage and food by imparting sweetness to them with the lactitol monohydrate crystals of the

present invention and the honey-containing crystals containing the same, because they are difficultly digested and absorbed as in other lactitol or lactitol crystals.

Therefore, the lactitol monohydrate crystals of the present invention and the honey-containing crystals containing the same can be utilized as a low calorie sweetener for patients with diabetes or fat people, and for sweetness tasting in low calorie beverage and food, e.g., beauty-mind food, healthy food, diet food, etc.

Furthermore, the lactitol monohydrate crystals of the present invention and the honey-containing crystals containing the same have properties that they are selectively utilized by Bifidus bacteria and are fermented with caries-inducing bacteria only with difficulty. Therefore, the crystals of the present invention may also be utilized as an agent for proliferating and activating Bifidus bacteria or as a sweetener which causes caries with difficulty.

The lactitol monohydrate crystals of the present invention and the honey-containing crystals containing the same are suitable for sweetness tasting in low caries food and beverage, for example, candies such as chewing gum, chocolate, biscuit, cookie, caramel, candy, etc.; beverage such as coke, cider, juice, coffee, lactobacillus beverage, etc.

The lactitol monohydrate crystals of the present invention and the honey-containing crystals containing the same well harmonize with a variety of substances which have other tastes such as sour, salty, astringent, tasty or bitter taste. In

addition, the crystals of the present invention have good resistance to acid and heat. Therefore, the crystals of the present invention can be freely used not only in the special cases stated hereinabove but also for sweetness tasting or taste improvement in ordinary food and beverage and for the purpose of improving the quality, etc.

The crystals of the present invention can be freely used in various seasonings such as soy sauce, soy sauce powder, miso, miso powder, Moromi or unrefined soy, Hisio or salted meat, various fish flour, mayonaise, dressing, vinegar, Sanbaisu or sauce of sake, soy and vinegar; vinegar for Sushi, formulated Chinese sauce powder, sauce for Tempura, sauce for Japanese noodle, sauce, ketchup, Korean sauce for baked beef, curry roux, instant stew mix, instant soup mix, instant soup stock, seasoning premix, nucleic acid type seasoning, sweet sake, synthetic sweet sake, table sugar, coffee sugar, fondant, etc.

The crystals of the present invention may also be freely utilized as a sweetener for various food and beverages such as Japanese cakes, e.g., Senbei or rice cracker, Arare or rice cake pellets, Okoshi or millet-and-rice cake, Mochi or steamed rice cake, Manju or bun with a bean jam filling, Uiro or steamed rice cake bun with a bean jam filling, bean jam, Yokan or sweet jelly of beans, cotton candy, jelly, sponge cake, candy, etc.; western cakes such as bread, biscuit, craker, cookie, pie, pudding, butter cream, custard cream, custard cream puffs, waffle, sponge cake, doughnut, chocolate, chewing gum, caramel, candy, etc.; fruit syrup, ice honey syrup; pastes such as flower paste, peanut

paste, fruit paste, etc.; processed fruits and vegetagles such as jam, marmalade, fruit syrup, fruit sugar candy, etc.; processed food of crops such as bread, noodle, rice, artificial meat, etc.; pickles such as Fukujinzuke or sliced vegetables pickled in soy sauce, Bettarazuke or fresh radish pickles, Senmaizuke or sliced fresh radish pickles, shallot pickled in soy sauce, etc.; instant mix for such as premix for Takuanzuke or pickled radish, premix for Hakusaizuke or Chinese cabbage pickles, etc.; meat paste products such as ham, sausage, etc.; fish paste products such as fish ham, fish sausage, steamed fish paste, broiled fish paste, fried fish paste, etc.; various table luxuries such as salted preserves of sea urchin or squid, Sukonbu or seaweed seasoned with vinegar, cut smoked squid, Fugu-no-mirinboshi or dried puffer seasoned with sweet sake and soy sauce, etc.; Tsukudani or food boiled down in soy, made of seaweed, edible wild plants, Surume or dried cuttlefish, small fish, shellfishes, etc.; cooked food such as cooked beans, potato salad, cooked rolled seaweed, etc.; bottled or canned food of milk products, fish meat, meat, fruit, vegetables, etc.; alcohols such as synthetic sake alcohol, fruit alcohol, western alcohol, etc.; beverages such as carbonate beverages, lactic acid beverages, etc.; premixes such as pudding mix, cake mix, etc.; instant beverages and food such as instant juice, instant coffee, instant Shiruko or adzuki bean soup, instant soup, etc.; or agents for improving taste and quality, etc.

Furthermore, the lactitol monohydrate crystals of the present invention and the honey-containing crystals containing

the same are hygroscopic only with difficulty and have good fluidity. Therefore, in chewing gum or Sukonbu or seaweed seasoned with vinegar, the crystals of the present invention may also be advantageously utilized as agents for preventing adhesion between the surface of the content and its packing paper or as agents for improving slippery, by coating the surface with the crystals.

Also for feeding animals such as livestock, chicken, bee culture, silkworm, fish, dog, cat, etc., the lactitol monohydrate crystals can be used for the purpose of improving the taste and quality or for the purpose of proliferating and activating Bifidus bacteria in animals.

In addition, the lactitol monohydrate crystals of the present invention can be freely used, in the form of solid, paste, liquid, etc., as agents for improving or controlling taste and further for improving quality in tobacco, tooth paste, lipstick, lip cream, drugs for internal use, troche, liver oil drop, stomatic refrigerant, stomatic flavor tablet, gargle, etc.

Furthermore, the lactitol monohydrate crystals of the present invention or the honey-containing crystals containing the same may be slightly wetted and subjected to compression molding under low pressure, thereby to freely mold into various shapes such as cube, fish, flower, etc., as in molded sugar prepared from granulated sugar. Therefore, molded sweeteners suitable for coffee, tea, etc. can be readily prepared.

In this case, the molding may be freely performed by incorporating various sugars, artificial sweeteners, etc. to

increase sweetness; or after coloring with various edible pigments or incorporating various flavors.

Upon use of flavors, various flavors may also be previously enclosed in clathrate compounds such as cyclodextrin, etc.

Further in the lactitol monohydrate crystals of the present invention, large crystals can be easily collected as in sugar so that they may be utilized as transparent or opaque non-hygroscopic sweeteners like ice sugar, coffee sugar, etc.

Furthermore, after the lactitol monohydrate crystals or the honey-containing crystals containing the same may be mixed with, for example, vitamins, antibiotics, lactic acid bacteria, etc., the mixture is prepared into various shapes such as granules, tablets, etc., which may be provided for various use.

(Examples)

Hereafter the present invention is described more specifically with reference to the examples below but is not deemed to be limited thereto.

In the following examples, % is all by weight unless otherwise indicated.

Example 1

After 80 g of a lactitol aqueous solution having a purity of 99.9% was charged in a laboratory plastomill, its concentration was adjusted to 88%. The lactitol aqueous solution was kneaded at a temperature of 40 °C for 10 minutes at 40 rpm to give about 75 g of the honey-containing crystals containing the lactitol monohydrate crystals of the present invention. A melting point of the crystals was determined as to whether a sample melted or

not, by putting the sample on a thermostat plate set forth at respective temperatures. As the result, the melting point was 103.5 °C.

The crystals were hardly hygroscopic, melted at a suitable temperature and were prepared easily. Therefore, the crystals can be advantageously used as sweeteners or agents for improving taste for various beverages and food, cosmetics, drugs, etc.; and further as raw materials for chemical industry.

Example 2

After 840 g of a lactitol aqueous solution (lactitol purity of 99.8%) having a concentration of 84.0% was gradually cooled from 130 °C, 0.2 g of seed crystals (obtained in Example 1) were added to the lactitol aqueous solution at a temperature of 45 °C. The mixture was allowed to cool over 20 hours to give a mass kit.

The mass kit was centrifuged to give 443 g of crystals (wet weight) and 458 g of the filtrate having a concentration of 69.2%.

The crystals were dried at a temperature of 50 °C for 18 hours using a net drier to give the lactitol monohydrate crystals of the present invention. In a manner similar to Example 1, a melting point of the crystals was determined to be 105.0 °C.

The results of elemental analysis are:

Calcd. C = 39.8%, H = 7.2%, O = 53.0%

Found C = 39.6%, H = 7.3%, O = 53.1%

Furthermore, differential scanning calorimetry (hereafter sometimes abbreviated as DSC) was performed. The results

indicate that the maximum endotherm was 107 °C at a temperature elevation rate of 10 °C/min.

Example 3

After 72 g of a lactitol aqueous solution having a purity of 99.9% and having a concentration of 90% was charged in a laboratory plastomill, 18 g of the honey-containing crystals obtained in a manner similar to Example 1 was added to the solution at a temperature of 50 °C. The mixture was kneaded at a temperature of 40 °C for about 3 minutes at 40 rpm to give the honey-containing crystals containing the lactitol monohydrate crystals of the present invention. A melting point of the crystals was determined as in Example 1. As the result, the melting point was 105 °C and the maximum endotherm by DSC was 104 °C.

Example 4

After 452 g of a lactitol aqueous solution (lactitol purity of 99.8%) having a concentration of 70.0% was gradually cooled from 130 °C, 0.2 g of seed crystals (obtained in Example 1) were added to the lactitol aqueous solution at a temperature of 30 °C. The mixture was allowed to cool over 20 hours to give a mass kit.

The mass kit was centrifuged to give 145 g of crystals (wet weight) and 262 g of the filtrate having a concentration of 55.6%.

The crystals were dried at a temperature of 50 °C for 18 hours using a net drier to give the lactitol monohydrate crystals of the present invention. In a manner similar to Example 1, a melting point of the crystals and the maximum endotherm were

determined to be 104.8 °C and 106 °C, respectively.

Example 5

After 20 kg of a lactitol aqueous solution having a purity of 99.0% and a concentration of 88.1% was gradually cooled to 35 °C, the solution was transferred to a stainless vat and 40 g of the honey-containing crystals obtained in a manner similar to Example 1 was added to the solution to thoroughly disperse. The mixture was allowed to stand at room temperature overnight to give a block mass.

Then, the block mass was ground into powders to give the powdery honey-containing crystals containing the lactitol monohydrate crystals of the present invention.

A melting point and the maximum endotherm of the crystals by DSC were determined in a manner similar to Example 1. The results are 103.5 °C and 104 °C, respectively.

Example 6

After 100 parts by weight of the lactitol monohydrate crystals or the honey-containing crystals containing the lactitol monohydrate crystals obtained in a manner similar to Example 3 and 1 part by weight of saccharin were homogeneously mixed, a small quantity of a lactitol aqueous solution having a concentration of about 75% was sprayed onto the mixture to render it wet. The mixture was charged in a mold for square sugar to mold under pressure. Then, the mold was withdrawn to give a solid sweetener composition molded into a cube.

The composition has sweetness of about 1.8 times that of sugar, is hardly hygroscopic and has good preservability. The

composition is caries-free sweetener which has substantially low calorie.

Example 7

After 40 parts by weight of cacao paste, 10 parts by weight of cacao butter, 50 parts by weight of the lactitol monohydrate crystals obtained in a manner similar to Example 2, 0.5 part by weight of aspartem and 0.5 part by weight of lecithin were mixed, the mixture was ground into fine powders by a refiner. The powders were put in a container and kneaded at a temperature of 50 °C for 24 hours.

Then, while cooling to 31 °C, the mixture was flown into a mold and solidified at 10 °C.

The product has not hygroscopicity and is useful as low caries chocolate having sweetness of good quality.

Example 8

After 50 parts by weight of aspirin, 15 parts by weight of the lactitol monohydrate crystals or the honey-containing crystals containing the lactitol monohydrate crystals obtained in a manner similar to Example 3 and 4 parts by weight of corn starch were thoroughly mixed, the mixture was prepared into tablets having a thickness of 5 mm and a diameter of 6 mm, using a tableting machine.

The tablet has no hygroscopic property and has sufficient physical strength, showing good degradation in water.

Comparative Example 1

(1) Preparation of comparative crystals

According to a modified process described in J. Agricultural

and Food Chemistry, 27, 4 (1979), 680, lactitol 1 for comparison was prepared and used for the following taste comparison test.

(2) Comparison test

As tasting panel, 20 male and female volunteers of 20 to 45 years old were joined the taste comparison test between the product prepared in Example 2 and the comparative crystals. The results are shown in the table below.

(scoring is as follows: 1 point where sweetness is pleasant, 0 point where sweetness is not pleasant, point 1 where sweetness is accompanied by bitter taste and 0 point where sweetness is not accompanied by bitter taste)

Item Compared	Pleasant	Bitter
Kind of Lactitol	Sweetness	Taste
Example 1	18	0
Comparative Example 1	2	16

(Effects of the Invention)

As described above, the lactitol monohydrate crystals of the present invention and the honey-containing crystals containing the same are useful as various food and drugs, etc. By practicing the present invention, these crystals can be prepared advantageously from an industrial viewpoint.

Brief Description of the Drawing

Fig. 1 is X ray analysis on crystalline structure showing single crystal of the novel lactitol monohydrate crystals of the present invention.